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OFFICE OF NAVAL RESEARCH

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MATERIALS RESEARCH IN COPENHAGEN AND ENVIRONS

HERBERT HERMAN

18 OCTOBER 1976

UNITED STATES OF AMERICA

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SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

REPORT DOCUMENTATION PAGE

READ INSTRUCTIONS BEFORE COMPLETING FORM

1. REPORT NUMBER 14 ONRL-R-8-76	2. GOVT ACCESSION NO.
3. RECIPIENT'S CATALOG NUMBER	
4. TITLE (and Subtitle) 6 MATERIALS RESEARCH IN COPENHAGEN AND ENVIRONS.	5. TYPE OF REPORT & PERIOD COVERED
	6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s) 10 HERBERT/HERMAN	8. CONTRACT OR GRANT NUMBER(s)
9. PERFORMING ORGANIZATION NAME AND ADDRESS Office of Naval Research Branch Office London Box 39, FPO New York 09510	10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS 12 14p.
11. CONTROLLING OFFICE NAME AND ADDRESS	12. REPORT DATE 11 18 Oct[redacted]76
	13. NUMBER OF PAGES 12
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office) 9 Conference rept.,	15. SECURITY CLASS. (of this report)
	15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

APPROVED FOR PUBLIC RELEASE; DISTRIBUTION UNLIMITED

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Materials science	Computer simulation
Denmark	Radiation Damage
Copenhagen	Nuclear materials

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Three materials science activities in Denmark were visited to update a report written by another liaison scientist some nine years ago. Two of the institutions are academic, The Technical University of Denmark at Lyngby, a suburb of Copenhagen, and the other directly in town, at the University of Copenhagen. A government laboratory was visited as well, The Research Establishment Risø, located north of the city. The development of science, especially materials science, in the midst of the evolving influences of social democracy, is discussed.

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S/N 0102-014-6601

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Materials Research in Copenhagen & Environs

About nine years ago a predecessor Liaison Scientist, J.B. Cohen, reported on the materials science situation in Denmark as part of an interesting review on materials research in Scandinavia (ONRL-40-67). In that report there were two institutions listed, whereas currently there are three that require consideration. Much has happened in the last few years which has influenced the development of science and technology within Denmark. Materials science has seen some very real growth and some problems as well. But to appreciate the situation *vis a vis* materials, it might be well to review some of the changes in academia and the sciences from which has emerged a "new order of politics."

About six years ago the Danish Ministry of Education declared that all educational and scientific establishments should be "promptly democratized." That is, from that day on, they should be administered by the will of the staff. This, of course, did not come out of nowhere, but had been brewing for some time. The university system of Denmark, in the tradition of old Teutonic authority, had been administered by the "empire system," with the "Professor" enunciating policy. In addition although this does not appear to have changed much, higher education was extremely drawn-out, normally taking over five years to achieve the first degree--with PhD's being very rare. Upon this environment was superimposed the general love of the Dane for freedom--which was then, and is now, giving rise to frequent political *causes célèbres*.

Into such a situation was thus introduced the concept of home rule for departments within the government-supported institutions of learning and study. The result was predictable. In some institutions, anarchy reigned--and does still today. In others, after minor changes (such as a listing of staff in alphabetical order instead of by the usually accepted heirarchy), the situation is more or less back to normal. But the net result of the upheaval of these recent years is that politics has entered the scene; in fact, I was told by several faculty members that the work of teaching and research has been generally, and in some areas of study, irreparably damaged. It seems that any issue can be brought up for a vote--and, last but not least, Department chairmen are now elected by the entire staff, including the secretaries, mechanics, technicians and students. One must be a sharp pol to survive in such a system. I was told by several faculty members that travel, always the life-blood of Danish science and scientists, is now a luxury, and one must be very careful about leaving campus, for when the cat's away....

However, it is the area of energy and its connected scientific issues which best exemplifies the present Danish political problems. It seems that Denmark is an extremely energy-poor country. Some 90% of its energy comes from imported oil, and virtually 100% of that from the Middle East. In fact, its luck has been poor in the international drilling scramble--Denmark's North Sea exploration fields, dry still, are adjacent to the rich finds of Norway. Thus, in 1956, the Research Establishment Risø, in Roskilde, of the Danish Atomic was established. It was the responsibility of this laboratory to devise methods and controls for the implementation of the country's future nuclear power program. In this they have been successful, and if all goes well, with careful planning and forethought the first power plant will be functioning by 1980. Now enters what in some quarters is called responsibility and in others, anarchy. Consistent with the mood of disestablishing the establishment, anti-nuclear groups have evolved--just as in most advanced countries of the world--and they are seriously threatening the schedule of Denmark's nuclear energy future.

Things have actually developed to the extent that in September the Danish Parliament will consider a move to place before the electorate a California-style nuclear referendum. This act, if instituted, could undermine and, perhaps, even destroy Denmark's attempt to improve its economy and cut down on its extremely high level of unemployment. It is interesting to see joint demonstrations against both nuclear energy and unemployment.

Enter the WIND at Tvind, where amateur windmill builders are constructing a 50 m high concrete tower which will presumably be the "world's tallest windmill." This windmill, built by committee, consistent with the "new democracy," is a direct and openly techno-political answer to the developing nuclear energy program. The designer and workers have had no experience in such construction projects, and I was told by reliable engineering authorities that the government won't touch it--principally for safety reasons. It seems that this answer to the danger of nuclear energy is being constructed in the midst of a schoolyard. Meanwhile this and similar projects continue to draw a great deal of attention in Denmark, and in other parts as well; the *New Scientist*, a magazine of UK origin, in the 10 June 1976 issue, features the Tvind project as a cover story. The article is an elementary and rather one-sided view of a most complex engineering project--and contains what are really hand-waving estimates showing that the wind and the nucleus are competitive. The final line of the *New Scientist* article perhaps

tells it best: "Meanwhile, as the energy debate rages in Denmark, the Tvind windmill stands as a defiant *fait accompli* for soft (sic) technology supporters everywhere."

It was in this land of academic and scientific excellence I sought the activities of materials engineers and scientists. Theirs were mixed moods of hopes and bits of depression. The students are good, but their numbers low, and as always these days--everywhere--there was a lack of research support. I visited three institutions: The Technical University of Denmark, The University of Copenhagen, and The Research Establishment Risø. These are extremely good laboratories, engaged in programs of excellence. It will be of some interest to see how that they evolve in future years in the present academic climate.

Technical University of Denmark

A few expressway miles to the north of Copenhagen is the Danish suburbia of Lyngby in which is located the institutes and laboratories of the Technical University of Denmark. The physics activities are comprised of 10 groups, one being materials science under the name of the Department of Structural Properties of Materials. This institute, which was founded about eight years ago by Prof. R.N.J. Cotterill, has emerged as the central materials science effort within the Technical University and very likely as the main department of the type in Denmark. Nine years ago, during his visit to Lyngby, Cohen reviewed the plans of metallurgy. At that time, Cotterill (who had formerly been with Hirsch and company at the Cavendish Lab) was a visiting scientist on leave from Argonne National Laboratory, engaged in point-defect work. He was given a professorship at the Technical University in 1968, and proceeded to establish an activity in materials science. There are actually two activities in materials--one metallurgy, of a more applied sort, and the other materials science. Similar situations, it happens, exist in several educational institutions the world over. In fact, in Denmark, as in most places where the two activities cohabit a campus, materials science seems to be "more equal." This may be due to a combination of more faculty talent, more student interest, more research support, etc. Such is the case at the Technical University, where metallurgy has ceased being visible, but Cotterill and his group, though small in number, have become successful and have had a real impact on the materials science of Denmark.

An additional aspect of Cotterill's leadership is that he is successfully able to couple with other groups on campus. Thus, there are joint research programs with: Prof. A. Thölen on electron microscopy of crystal imperfections; Prof. G. Trumpy on positron annihilation studies; Prof. A. Lindegaard Andersen on x-ray studies of elasticity and defect configurations. Also, the Department collaborates with the several groups within the Danish Research Establishment Risø.

Educationally, the Department is responsible for administering a degree of "civilingeniør" (MS), which is obtained after five and a half years of study, and "lic. techn." (PhD), which normally requires a further two and a half years of study and research. (If you've ever wondered why you haven't heard from young Danish scientists looking for jobs, a good reason may be that the graduating "civilingeniør starts at about the equivalent of $15,000--and if they become "redundant," I was told that welfare payments exceed salaries of most European countries.)

The teaching program appears for the most part to be standard materials science fare, but due to Cotterill's interest in the computer, there is an undergraduate lecture/laboratory course on computer simulation. This hands-on course uses an IBM 360/75 together with peripheral plotting equipment. The students are taught programming and learn techniques for the simulation of the perfect crystal and calculations of interatomic potential. Perfect and imperfect crystals are studied using lattice statics and molecular dynamics.

Cotterill has been continuing his research on melting. He has, in fact, together with E.J. Jensen, W.D. Kristensen, R. Paetsch and P.O. Esbjørn (Risø), developed a unified theory of melting, crystalization and glass formation. These ideas arose from his pet activity which has been computer-modeling of defects in solids. The melting studies began several years ago when he was investigating the melting of a microcrystal in two dimensions using molecular dynamics simulation. Since these initial results, the program has evolved into research in the currently popular field of amorphous alloys.

Cotterill's view of melting, as obtained by computer simulation, is that the phenomenon is initiated by the nucleation of an instability at the locus of dislocations which come together in sufficient density. This is not a new idea, having previosly been proposed by N.F. Mott, W. Schockley and others, but in Cotterill's hands the concept has taken on a new form. I was pleased to see their computer movie of the melting process, and, though there are still uncertainties and room for debate, the movie is an exercise in discovery and is rather exciting. Put simply, as the temperature of the crystal approaches the melting point, the vibrational amplitudes become so large that bond rearrangements can occur. The computer shows that the rearrangements give rise to slipping, with the result that dislocations are formed in pairs or dipoles.

From their computer simulation schemes, extended to three dimensions, Cotterill and co-workers have found excellent agreement, for Lennard-Jones crystals, with the observed melting point, volume change and latent heat. The three-dimensional dipole arrangement *actually generates the correct liquid-like pair-distribution function.* The concept is appealing and has been developed rather far. In fact, on the experimental front, the laboratory is attempting to observe melting directly in the electron microscope. Initial results are promising. They have examined the melting of aluminum (imbedded between oxide sheets) *in situ* within the electron microscope. According to Cotterill, there is clear evidence in this work that melting is initiated by a dislocation mechanism.

It may be obvious that the currently active area of amorphous alloys would be a good place to test Cotterill's ideas of the dislocation-structure of liquids and melting. And, indeed, the group is doing just that. Not by electron microscopy, but rather with *field-ion microscopy(!). Using a commercial instrument,* they have successfully imaged "lattice spots" from sharpened tips of splat-cooled Pd-Si glass. This important experiment may pave the way towards a direct approach to understanding the structure of glasses. This work has been underway for only a short time. The only difficulty encountered, thus far, was the pointing of the target which was achieved by alternatively polishing and etching the specimen. Other work is underway with this alloy-glass system, including a scanning electron-microscope (SEM) study of fracture surfaces.

In other words, taking Cotterill's lead in computer simulation, point defects are still being studied in close-packed crystals and

more complex systems, such as ice. Further, experimentally, there is activity with other departments on impurity-vacancy interactions in metals (e.g., in aluminum with K.O. Sørensen) and on positron annihilation at defects in crystals. Cotterill's work has been extremely visible in this latter field, and together with a very able positron group at Lyngby, he has been able to employ this technique to study defects in crystals. In fact, he will present a review of defect studies at the September Positron Annihilation Conference at Lyngby. Cotterill firmly believes that positrons are not trapped by dislocation cores, or by interstitials, but by vacancies only. He thus feels that if defects are detected following plastic deformation or fatique, one is really "seeing" jogs. An associate, J.G. Rasmussen, is using positron annihilation to study fatique. He is examining the development of primary and secondary slip bands containing intrusions and extrusions at the surface of aluminum single crystals. These observations are being related to changes in the positron spectra.

There are a number of other interesting research programs underway within Cotterill's group. For example, in collaboration with workers at Risø, metals near their melting points are being studied with neutron diffraction. Pre- and post-melting effects are being sought. Some early work on solid aluminum at 0.01°C from the melting temperature shows no softening of lattice vibrational modes, indicating no evidence of pre-melting phenomena. Liquid crystals and their phase transitions are being studied by E.J. Jensen. The molecular dynamics technique is being used to study how different interatomic potentials and molecular geometries affect the number of mesophases exhibited by the system.

Cotterill is also applying the molecular dynamics approach to yet another field--biophysics. This, he relates, has been a long-time interest. And, finally, he has begun examining defects and phase transitions in phospholipid membranes. The characteristic behavior of the membrane surface is known to be related to phase changes which are induced by temperature and pH change. The membrane system is represented by a quasi two-dimensional structure, with a complex intermolecular potential scheme being required. Using the computer, Cotterill has detected an influence of temperature and electrical screening on the two-dimensional order-disorder transition (related to the diffusive properties of the membrane). He has evidence that the transition is initiated by the spontaneous generation of disclinations. Considerable interest in Cotterill's work in this field is developing in Europe.

It is clear that the Department of Structural Properties of Materials has a number of first-rate people engaged in very topical

problems. Cotterill has given excellent and stimulating leadership. However, student interest in materials science is low. Much work is being done by the Department to strengthen this interest on campus. Added to the problem, the University is clearly overbuilt (with 3000 students, having originally been built to house 10,000).

<u>University of Copenhagen</u>

The solid state research unit of the Physics Laboratory II, H.C. Ørsted Institute, is housed in a newish building in the northern section of Copenhagen. There is an interesting activity underway in radiation effects on materials. L.T. Chadderton, who came here in 1970 as professor, has formed a group dedicated to studying a wide range of the materials science aspects of radiation effects. He has programs in electron damage in alkali halides, ion implantation, sputtering--at the Institute's laboratories and cooperatively with workers at Aarhus University and rather more internationally. A new senior staff member, Dr. J.L. Whitton, who joined Chadderton last October after 15 years at Chalk River in Canada, is beginning a large program on ion-implantation and channeling.

From the outset, Chadderton's period as chairman has been an up-hill job, the Ørsted Institute being historically and geographically related to biology and the medical schools of the University. Therefore, theoretical and experimental biophysics have always been big items. Chadderton faced this life-sciences orientation and--perhaps more importantly--the unstable period when the University, and the Physics Laboratory, began to undergo their period of "democratization." Those days, Chadderton explained, were trying, but there is finally a feeling that the Laboratory is out of the period and back to the job of building solid-state sciences, particularly studies of radiation effects, in a scope broader than is generally meant, in gases, glasses, etc., and using a wide range of radiation. It appears that Chadderton is achieving his goal.

To assist in the development of the Laboratory, Whitton has already begun programs on ion implantation, channeling and surface physics. Together with Chadderton and H.A. Johansen, he has been looking at the annealing of silicon following radiation damage induced by the ion implantation of 80-keV Cu^+, Au^+ or Au^+ ions up to doses of 10^{15} ions/cm^2. Transmission electron microscopy (TEM) and Rutherford backscattering (RBS) are used for this program. Silicon implanted with Au^+ shows damage-recovery on annealing to temperatures up to 800°C, whereas Cu^+-and Ag^+-induced damage remains (as measured by

RBS). There is clear evidence that for Au^+, diffusion to the surface occurs. There is good evidence from TEM that for the three ions, an amorphous/polycrystalline transition occurs. Whitton is also using channeling and RBS for something metallurgical--to detect precipitation in dilute Nb-H and Nb-D alloys. The backscattered 1-MeV He ions which channel along $\langle 100 \rangle$ of Nb-H crystals are sensitive to the microstructure, and the backscattered yield will depend on the presence of a second phase. This very sensitive technique, which is able to detect phase changes, has been used here to help fill in the phase diagram (at low compositions and low temperatures). They have been working with KFA, Jülich, West Germany, and have studied alloys in the concentration range 0.24 to 3.45 at.% from 161 to 249 K. Further studies using this approach are continuing in the systems V-H, V-D, Ta-H, Ta-D and Zr-H.

Further on the subject of radiation effects, Whitton is studying the surface topography of Cu bombarded by 40-keV Ar ions. This work, which is being done cooperatively with J.S. Williams of the University of Salford (UK), is investigating the formation and growth of macroscopic geometric cones observed by SEM following heavy-ion sputtering. This effect is believed simply to be due to an orientation-dependent "sputterability," but the details of the development of this beautiful surface structure are anything but simple.

Chadderton is continuing his work on channeling, with special attention being given to the details of directional radiation effects in crystals. He is looking at quasi-channeling and techniques for determining atom locations.

A very interesting program on void formation in irradiated fluorite is being carried out by Chadderton, E. Johnson and T. Wohlenberg. It appears that when fluorite (calcium fluoride) is electron-irradiated within the electron microscope (at 100 keV) a simple cubic void superlattice is formed. This discovery by these workers is being explored in some detail, from both a structural and a kinetic viewpoint. The voids which are seen on irradiation are believed to arise from bubbles of fluorine gas (giving rise to loop contrast in TEM) which comes from the radiation-induced chemical reaction $CaF_2 \rightarrow Ca + F_2$. Diffusion, it is supposed, occurs on the anion sublattice only, by way of a crowdion mechanism, and the Ca/CaF_2 misfit parameter is the principal factor in the establishment of a void superlattice. Chadderton explained that although this situation is somewhat different from that which exists in metals, these experiments may contribute to the general problem of void formation in irradiated crystals. In fact, he feels that "in spite of this defect's current unpopularity, ... a fundamental reassessment (is needed) of the part played by the crowdion in void lattice formation in metals."

Another area of great interest to Chadderton is the study of low-energy electron reflection from surfaces by examining the "total current spectra" (TCS). The experimental situation is straightforward: using a modulation technique, he measures the sample current as a function of electron energy, E_1. This energy is varied by changing sample potential to make the primary beam current independent of energy. The sample's total electron current is the difference between the primary and secondary currents, and the main dependence of energy on total current is thus contained in the secondary current. The TCS curve gives information on electron-solid interactions, and it is thought here that in many ways these can be direct experimental probe for studies of solid surfaces.

In the more traditional areas of radiation effects, Chadderton and Johnson are studying heavy-ion-irradiated zinc crystals with TEM and RBS. They are especially interested in RBS experiments in hexagonal metals having low Debye temperatures. Zinc ions are being implanted into the crystals, and TEM is used to study the basal planes, which contain most of the crystallographic defects. This sort of crystal will then be studied with RBS, in order to attempt an assessment of the effects of a known distribution of defects on the RBS spectra.

Johnson (in a joint program with Sir Peter Hirsch of Oxford) is examining *in situ* deformation behavoir in HVEM of neutron irradiated copper single crystals. The deformation of the irradiated crystals (to doses of 10^{18} n/cm^2, $E > 1$ MeV) is heterogeneous. Microscopic observation shows that dislocation motion is confined to the slip bands, with only screw-oriented dislocations experiencing motion. The moving dislocations sweep up point defects, giving rise to jogged lines. Further straining causes bowing between the main points, and hence a large number of dipoles are formed.

The Laboratory is engaged in a number of other studies, both theoretical and experimental, over a fairly wide range of solid-state physics topics. The materials science here is concerned mainly with physics and less so with technological materials. This is a well-outfitted laboratory, receiving good support and, like most Danish scientific organizations, very much coupled to the outside world.

The Laboratory staff is listed below:

Laboratory Personnel (as of Summer, 1976)

L.T. Chadderton - Professor
N.O. Andersen
S.A. Andersen
B. Buchmann
B. Buras
F.E. Carlsen
H.A. Johansen
E. Johnson
J.S. Olsen
L. Sarholt-Kristensen
H.P. Sigmund
S. Steenstrup
N.E.W. Veje
J.L. Whitton
T. Wohlenberg

Research Establishment Risø

The Research Establishment is at Roskilde, some 25 miles north of Copenhagen, and is situated on a 600-acre site, partly on the beautiful Roskilde Fjord. The laboratory started life in 1956 as the Danish Atomic Energy Commission Research Establishment, but very recently has been taken over by the Ministry of Trade and Industry and, though much of the work is still nuclear, the designation will now be "Research Establishment Risø." I met with Dr. N. Hansen, head of the Metallurgy Department, and a number of the staff. The work is fairly broad, running from applied metallurgy to materials science research, with a rather strong emphasis on the former. What has motivated Risø generally, and Metallurgy in particular, are Denmark's plans to go nuclear by 1980--politics aside. Risø, then, is becoming the local scientific and engineering authority for the future and, as such, has developed programs in design and fabrication of fuel elements, testing of nuclear components, structural and mechanical studies, radiation damage and corrosion. There are three experimental reactors (2-kW and 5-MW light water reactors and a 10-MW heavy water reactor). In addition, there are three accelerator facilities and hot cells.

A long-term study on dispersion-strengthened $Al-Al_2O_3$ continues. This work, which is under the direction of Hansen, has concentrated on recrystallization behavior with special reference to the effects of particle size. The recrystallization temperature is observed to increase rapidly with increasing particle size for small particles (starting at 360 Å) and decreases for larger particle sizes (to 850Å)--for an almost constant particle spacing of 0.4 µm. Hansen explains these results by the superposition of a retarding effect (for small particles) and an accelerating effect (for larger particles). The small-particle effect is related to delayed dislocation-climb around the particles, whereas the accelerating

comes about from the formation, during recovery, of small misoriented areas around the particles.

N. Kjaer-Pedersen has developed a model for predicting UO_2-Zr performance, based on the superposition principle in a finite difference description of the pellet elastic behavior and shell theory. A power history is simulated for one segment of a fuel rod, with creep being incorporated as well. A 3-dimensional representation of fuel cracking is thus achieved, making this a very important technique of evaluation.

T. Leffers has worked on grain size and rolling texture in Cu-Sn alloys and, with Cotterill (Danish Technical University) and H. Lilholt, on a molecular dynamics approach to grain boundary structure and migration. Using Cotterill's computer simulation technique, they have demonstrated that grain boundary formation from the melt can be described by the molecular dynamics method.

Leffers and B.N. Singh are engaged in an interesting study of Cu-Ni and its behavior in a radiation field. This work started, in fact, with stainless steel, containing particle-controlled grain refiners. This approach (grain refining) has generally been able to decrease swelling due to void formation. They have decided to broaden this sort of study by examining the effects of alloying on void formation in a radiation field--thus Cu-Ni, which is used as a "model system." The experiments are being done with Harwell's (UK) 1-MeV electron microscope, and concentrations up to 10 wt%Ni are being examined. Initial results indicate that void-induced swelling decreases with temperature of irradiation up to 450°C, but a peak is observed for low compositions at temeratures above 500°C. At 10 wt%Ni, on the other hand, the swelling approaches zero. Since dislocation loops are seen to be generated by "invisible" particles during the irradiations, these workers believe that this experiment, at least indirectly, demonstrates the presence of clustering for Cu-Ni. This seems to be the case since the void density increases with atom fraction of Ni, <u>but</u> the void volume decreases. The solute clusters (still unobserved) appear to act as nucleation centers for the voids. Since small-angle x-rays can't effectively resolve this situation (due to the closeness of the atomic scattering factors of Cu and Ni), it is clear that someone ought to look at this system with small-angle neutron scattering (assuming the differential cross sections differ sufficiently). In fact, Leffers and Singh are examining this possibility.

There is a considerable effort underway on non-destructive testing (NDT), with acoustic emission (AE) receiving some attention from a new man from the UK, W.E. Swindlehurst. Swindlehurst wants to use AE to study carbide fracture in steels. He wants particularly to determine whether carbide cracking can initiate fracture, and feels that AE is a good enough approach for this sort of problem. My impression was that Swindlehurst recognizes the limitations of AE, and many of the problems associated with the techniques, but feels that there is a future in AE when used properly.

NDT is actually big business at Risø, or at least they would like it to be. An automatic, high speed tube inspection system has been developed and patented by Risø. The device incorporates a rotating water chamber together with eight ultrasonic transducers. Nuclear fuel tubes can be inspected at speeds of 6 m/min with the chamber (not the tube!) rotating at 3000 rpm.

Further on the commercial front, P. Knudsen explained to me that Risø has developed a good working relation with Helsingør Vaerft A/S, a leader in Danish shipbuilding, and, more recently, in the nuclear business. They, together with workers at Risø, have started programs in fuel modeling, reactor fuel reprocessing and canning, and NDT. Thus Danish industry is preparing itself for the coming of nuclear power systems.

* * *

In conclusion, it is easy to note that the situation in Denmark is fluid--much depending on politics over a range of issues from social democracy to energy. The student problems appear to be decreasing in both frequency and in intensity, but another has taken its place--where to get good students interested in the materials sciences. This international problem has no easy answers, but Cotterill, for one, is working hard at attracting young people into his program. They University of Copenhagen, however, with a strong orientation and tradition in physics has found that it competes for physicists against other notable Danish physics academic institutes, so it will be hard work for some time.

Nevertheless, all things considered, materials science in Denmark is a going operation and, to quote my favorite travel guide, "Worth the detour."